16.1.3.4 Sulfate Analysis. Adjust the flow rate through the ion exchange column to 3 ml/min. Pipette a 20-ml aliquot of the filtrate onto the top of the ion exchange column, and collect the eluate in a 50-ml volumetric flask. Rinse the column with two 15-ml portions of water. Stop collection of the eluate when the volume in the flask reaches 50-ml. Pipette a 20-ml aliquot of the eluate into a 250-ml Erlenmeyer flask, add 80 ml of 100 percent isopropanol and two to four drops of thorin indicator, and titrate to a pink end point using 0.0100 N barium perchlorate. Repeat and average the titration volumes. Run a blank with each series of samples. Replicate titrations must agree within 1 percent or 0.2 ml, whichever is larger. Perform the ion exchange and titration procedures on duplicate portions of the filtrate. Results should agree within 5 percent. Regenerate or replace the ion exchange resin after 20 sample aliquots have been analyzed or if the end point of the titration becomes unclear.

NOTE: Protect the 0.0100 N barium perchlorate solution from evaporation at all times.

16.1.3.5 Blank Determination. Begin with a sample of water of the same volume as the samples being processed and carry it through the analysis steps described in Sections 16.1.3.3 and 16.1.3.4. A blank value larger than 5 mg should not be subtracted from the final particulate matter mass. Causes for large blank values should be investigated and any problems resolved before proceeding with further analyses.

16.1.4 Calibration. Calibrate the barium perchlorate solutions as in Method 6, Section 10.5.

16.1.5 Calculations.

16.1.5.1 Nomenclature. Same as Section 12.1 with the following additions:

m_a = Mass of clean analytical filter, mg.
m_d = Mass of dissolved particulate matter, mg.
m_e = Mass of beaker and dissolved particulate matter after evaporation of filtrate, mg.
m_p = Mass of insoluble particulate matter, mg.
m_r = Mass of analytical filter, sample filter, and insoluble particulate matter, mg.
m_{bk} = Mass of nonsulfate particulate matter in blank sample, mg.
m_n = Mass of nonsulfate particulate matter, mg.
m_s = Mass of Ammonium sulfate, mg.
N = Normality of $Ba(ClO_4)$ titrant, meq/ml.
V_a = Volume of aliquot taken for titration, 20 ml.
V_c = Volume of titrant used for titration blank, ml.
V_d = Volume of filtrate evaporated, 200 ml.
V_e = Volume of eluate collected, 50 ml.
V_f = Volume of extracted sample, 500 ml.
V_i = Volume of filtrate added to ion exchange column, 20 ml.
V_t = Volume of $Ba(C10_4)_2$ titrant, ml.
W = Equivalent weight of ammonium sulfate, 66.07 mg/meq.

16.1.5.2 Mass of Insoluble Particulate Matter.

$$m_p = m_r - m_a - m_f \qquad \text{Eq. 5F-4}$$

16.1.5.3 Mass of Dissolved Particulate Matter.

$$m_d = \left(m_e - \left(V_f / V_d\right) m_b\right) \qquad \text{Eq. 5F-5}$$

16.1.5.4 Mass of Ammonium Sulfate.

$$m_s = \frac{\left(V_t - V_c\right) N\, W\, V_e V_f}{V_a V_i} \qquad \text{Eq. 5F-6}$$

16.1.5.5 Mass of Nonsulfate Particulate Matter.

$$m_n = m_p + m_d - m_s - m_{bk} \qquad \text{Eq. 5F-7}$$

17.0 References

Same as Method 5, Section 17.0, with the addition of the following:

1. Mulik, J.D. and E. Sawicki. Ion Chromatographic Analysis of Environmental Pollutants. Ann Arbor, Ann Arbor Science Publishers, Inc. Vol. 2, 1979.

2. Sawicki, E., J.D. Mulik, and E. Wittgenstein. Ion Chromatographic Analysis of Environmental Pollutants. Ann Arbor, Ann Arbor Science Publishers, Inc. Vol. 1. 1978.

3. Siemer, D.D. Separation of Chloride and Bromide from Complex Matrices Prior to Ion Chromatographic Determination. Analytical Chemistry 52(12): 1874–1877. October 1980.

4. Small, H., T.S. Stevens, and W.C. Bauman. Novel Ion Exchange Chromatographic Method Using Conductimetric Determination. Analytical Chemistry. *47*(11):1801. 1975.

18.0 Tables, Diagrams, Flowcharts, and Validation Data. [Reserved]

METHOD 5G—DETERMINATION OF PARTICULATE MATTER EMISSIONS FROM WOOD HEATERS (DILUTION TUNNEL SAMPLING LOCATION)

NOTE: This method does not include all of the specifications (*e.g.*, equipment and supplies) and procedures (*e.g.*, sampling and analytical) essential to its performance. Some material is incorporated by reference from other methods in this part. Therefore, to obtain reliable results, persons using this method should have a thorough knowledge of at least the following additional test methods: Method 1, Method 2, Method 3, Method 4, Method 5, Method 5H, and Method 28.